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तैयार मिश्रित पेंट, परिष्करण, हवा में सूखने, युद्ध के उपकरणों के लिए — विशिष्ट

(दूसरा पुनरीक्षण)

Ready Mixed Paint, Finishing, Air-Drying for War Equipment — Specification

(Second Revision)

ICS 87.040

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Paints, Varnishes and related Products Sectional Committee, CHD 20

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Paints, Varnishes and Related Products Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1978 and subsequently revised in 1991. It was made on the request from Ministry of Defence and was based on JSS 8010-15 'Specification for paint, ready for use, finishing, war equipment, air drying, brushing/spraying, olive green, white and buff light, Indian Standard Colour No. 358'.

The first revision of this standard was published with a view to assimilate the technological advancements, which had taken place since its publication. In the first revision of the standard, requirements for some of the parameters such as wet opacity, gloss, fineness of grind, etc, were quantified. New parameters of accelerated storage/stability test were also incorporated to align the standard with the revised defence specification.

This (second) revision has been taken up with a view to incorporate the limit of lead restriction. The Committee responsible for formulation of this standard observed that such paints are also used for household/decorative purposes. Taking cognizance of the fact that lead exposure of humans, particularly children in residential premises, is injurious to health and has adverse impact on human health and safety, the Committee felt the need to introduce lead restriction in all Indian Standards on paints.

The Committee observed that the material is used for protection and decoration of metal parts for household/ industrial applications and is, therefore, accessible to common public. The Committee also observed that it is technologically feasible to formulate the material with low lead content. Therefore, in this revision, the requirement of maximum permissible limit of lead has been prescribed as 90 ppm along with the relevant method of test. This limit has been prescribed, keeping in view of the scope for exposure of children to the material after drying and stoving, and also aiming at avoiding hazardous impact of lead on environment and health of living beings.

Further, majority of consumers are not aware of the consequences of lead toxicity and its long term implications to human health. Therefore, in this revision, along with lead restriction, a suitable cautionary notice has also been included in the marking clause.

Reference has been given to various parts/sections of IS 101 for testing the requirements prescribed in this standard. This standard refers to IS 5 : 2007'Colours for ready mixed paints'.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

READY MIXED PAINT, FINISHING, AIR-DRYING FOR WAR EQUIPMENT — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for ready mixed paint, finishing, air drying, for war equipment. The material is used for protection and maintenance of wooden and metal parts of war equipment, including vehicles, machine guns, bridging equipment, etc and also on POL containers as exterior petrol resisting paint.

1.1.1 The material is intended for distinctive lettering and marking, with rubber stamps of containers, packages, ammunition and ammunition packages, where quick drying is desired. The material is not intended for use on ammunition and ammunition packages containing nitrophenolic explosives.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards:

IS Number	*Title*
5 : 2007	Colours for ready mixed paints and enamels (*sixth revision*)
101 (Part 1)	Methods of sampling and test for paints, varnishes and related products: Part 1 Test on liquid paints (general and physical) (*third revision*)
Sec 1 : 1986	Sampling (*third revision*)
Sec 2 : 1987	Preliminary examination and preparation of samples for testing (*third revision*)
Sec 3 : 1986	Preparation of panels (*third revision*)
Sec 5 : 1989	Consistency (*third revision*)
Sec 6 : 1987	Flash point (*third revision*)
Sec 7 : 1987	Mass per 10 litres (*third revision*)
Part 2	Test on liquid paints (chemical examination)
Sec 1 : 1988	Water content (*third revision*)
Part 3	Tests on paint film formation
Sec 1 : 1986	Drying time (*third revision*)
Sec 4 : 1987	Finish (*third revision*)
Sec 5 : 1987	Fineness of grind (*third revision*)
Part 4	Optical test
Sec 1 : 1988	Opacity (*third revision*)
Sec 2 : 1989	Colour (*third revision*)
Sec 3 : 1988	Light fastness test (*third revision*)
Sec 4 : 1988	Gloss (*third revision*)
Part 5	Mechanical tests
Sec 1 : 1988	Hardness test (*third revision*)
Sec 2 : 1988	Flexibility and adhesion (*third revision*)
Part 6	Durability test on paint films
Sec 1 : 1988	Resistance to humidity under conditions of condensation (*third revision*)
Sec 2 : 1989	Keeping properties (*third revision*)
Part 7	Environmental tests on paint films
Sec 2 : 1990	Resistance to liquids (*third revision*)
Sec 3 : 1990	Resistance to heat (*third revision*)
Part 8	Tests for pigments and other solids
Sec 5 : 1993	Lead restriction test (*third revision*)
1070 : 1992	Reagent grade water
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part 1 Dangerous goods
1303 : 1983	Glossary of terms relating to paints (*second revision*)
1407 :1980	Round paint tins (*second revision*)
1745 : 1978	Petroleum hydrocarbon solvents
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
13656 : 1993	Textile dyestuffs — Dispersibility of disperse dyes — Determination

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1303 shall apply.

4 REQUIREMENTS

4.1 General

The material shall be supplied in brushing consistency, but shall be suitable for application by spraying after thinning with petroleum hydrocarbon solvent, low aromatic, 145/205 grade (*see* IS 1745)

4.2 Colours

The colour of the material shall be white or scamic Indian Standard Colour No. 294 or light buff Indian Standard Colour No. 358, as required. It shall be close match to specified colour when tested as per IS 101 (Part 4/Sec 2). For match against Indian Standard colour, IS 5 shall be referred.

4.3 Pigment Composition

The dry pigment content of the material shall be 33 ± 3 percent by mass. In case of olive green paint, the pigment shall contain minimum 6 percent by mass of green oxide of chromium when determined as prescribed in Annex A.

4.4 Lead Restriction

The material shall be tested for restriction of lead in accordance with IS 101 (Part 8/Sec 5). When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead, exceeding 90 ppm.

4.5 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.6 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the paint shall be packed in metal containers (*see* IS 1407 and IS 2552).

5.2 Marking

5.2.1 Each container shall be marked with the following:

a) Name and colour of the material,
b) Indication of the source of manufacture,
c) Volume of the material,
d) Batch No. or Lot No. in code or otherwise, and
e) Month and year of manufacture.

5.2.2 Each container shall have a caution label **'HIGHLY FLAMMABLE',** together with the corresponding symbol for labelling of dangerous goods [*see* Fig. 3 of IS 1260 (Part 1)].

NOTES

1 Necessary safeguard against the risk arising from the storage and handling of large volumes of flammable liquids shall be provided and all due precautions shall be taken at all times to prevent accidents by fire or explosion.

2 Except when they are opened for the purpose of cleaning and rendering them free from vapours, all empty tanks or other containers shall be kept securely closed.

5.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

6 SAMPLING

6.1 Representative samples of the material shall be prepared as prescribed in IS 101 (Part 1/Sec 2) : 1986.

6.2 Criteria for Conformity

A lot shall be declared conforming to the requirements of this specification if the test results on the composite sample satisfy the requirements prescribed in **4**.

Table 1 Ready Mixed Paint, Finishing, Air-Drying, for War Equipment

(*Clause* 4.6)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS 101
(1)	(2)	(3)	(4)	(5)
i)	a) Consistency	Smooth and Uniform	—	(Part 1/Sec 5)
	b) Efflux time by Ford Cup No. 4 at 27 ± 2°C, *sec*	80-120		
ii)	Application	Suitable for application by brushing without appreciable drag on the brush	—	(Part 1/Sec 1)
iii)	Drying time:		—	(Part 3/Sec 1)
	a) Surface dry	Not less than 1 h		
	b) Hard dry and tack free	Not more than 8 h		
iv)	a) Finish	Smooth and semi-glossy	—	(Part 3/Sec 4)
	b) Gloss value, 45° (Specular gloss)	31 - 50 units	—	(Part 4/Sec 4)
	c) Fineness of grind, micron, *Max*	40	—	(Part 3/Sec 5)
v)	Wet opacity, *Min*, m^2/10 litre (for olive green only)	250	—	(Part 4/Sec 1)
vi)	Scratch hardness (1500 g)	Bare metal shall not be visible through the scratch after 48 h	—	(Part 5/Sec 2)
vii)	a) Flexibility and adhesion[1)]	No damage or detachment or cracking of the film	—	(Part 5/Sec 2)
	b) Flexibility and adhesion at 0°C (for white only)[2)]	No damage or detachment or cracking of the film	—	—
viii)	Protection against corrosion	There shall be not more than very slight change of colour not more than slight superficial rust spotting, no blistering of permanent nature (blistering which does not subside within 24 h after removal of the painted panel from the paint corrosion cabinet shall be termed as permanent), no brittleness of the paint film and the metal surface underneath the film shall not show any sign of corrosion	—	(Part 6/Sec 1)[3)]
ix)	Resistance to lubricating oil (*see* IS 13656)	No sign of permanent injury	—	**4** of 101 (Part 7/Sec 2)
x)	Resistance to petrol-benzol mixture	No sign of permanent injury	B	—
xi)	Resistance to heat (except white)	No sign of cracking, blistering and appreciable change of colour when stoved at 150°C for 2 h. In this respect the film shall not be inferior to registered sample	—	Part 7/Sec 3
xii)	Water absorption test	Amount of water absorbed shall not exceed 20 mg/cm^2 of painted surface exposed	C	—
xiii)	Resistance to warm water at 45 ± 3°C for 4 h.	No fading or change of colour	D	—
xiv)	Resistance to salt water	No rust spotting, flaking or peeling or any other deterioration of the paint film when examined at a magnification of 10 diameter	E	—
xv)	Durability	To pass the test	F	—
xvi)	Flash point, °C, *Min*	30	—	(Part 1/Sec 6)
xvii)	Calcium compounds in dry pigments (as CaO), percent by mass, *Max*	5	G	—
xviii)	Barium compounds in dry pigments (as BaSO4), percent by mass, *Max*	33	H	—

Table 1 (*Concluded*)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS 101
(1)	(2)	(3)	(4)	(5)
xix)	Water content (if water suspected to be present), percent by mass, *Max*	0.5	—	(Part 2/Sec 1)
xx)	Mass in kg/10 litres, *Min*	11.0	—	(Part 1/Sec 7)
xxi)	Accelerated storage stability test	To pass the test	J	—
xxii)	Keeping properties, *Min*	The material shall not cake hard inside the containers and shall retain properties for one year from the date of delivery	—	(Part 6/Sec 2)

[1)] The panel after application shall be stoved at 100°C for 1hafter initial drying of 24 h. The test shall be carried out after the panels are allowed to stand at room temperature not below 15°C for a further period of 24 h.

[2)] The panel shall initially be air dried at room temperature of not below 15°C for seven days and thereafter shall be kept along with the bend test apparatus at 0°C for 2 h.

[3)] Except for duration of the test period which shall be 10 days.

ANNEX A

(*Clause* 4.3)

DETERMINATION OF GREEN OXIDE OF CHROMIUM

A-1 GENERAL

Chromium oxide (Cr_2O_3) content of the pigment is estimated from acid insoluble chromium compounds. The method involves the estimation of total chromium compounds as Cr_2O_3 in pigment of the paint.

A-2 REAGENTS

A-2.1 Sodium Peroxide

A-2.2 Sulphuric Acid — Concentrated.

A-2.3 Potassium Permanganate Solution — 5 percent (*m*/*v*).

A-2.4 Silver Nitrate Solution — 10 percent (*m*/*v*).

A-2.5 Ammonium Persulphate

A.2.6 Hydrochloric Acid — Dilute.

A-2.7 Phosphoric Acid— Specific gravity 1.8.

A-2.8 Ferrous Ammonium Sulphate Solution — Approximately, 0.1 N.

A-2.9 Standard Potassium Permanganate Solution — 0.1 N

A-3 ESTIMATION OF CHROMIUM COMPOUNDS IN PAINT

A-3.1 Procedure

Weigh accurately about 1 to 2 g of the paint in a nickel crucible and ignite over a gas flame. Fuse it with 5 g of sodium peroxide over a gas flame, keeping the molten mass at low red heat for 5 min. Dissolve the cooled fusion cake in the crucible in 200 ml of warm water and transfer the rinsing to a beaker. Add concentrated sulphuric acid to adjust 10 percent acidity in the solution. Add 4 to 6 drops of 5 percent potassium permanganate solution and 1 to 2 ml of silver nitrate solution. Heat to boil. Add ammonium persulphate till pink colour of potassium permanganate reappears. Add dilute hydrochloric acid just to destroy the pink colour. Boil to expel chlorine. Cool and add 5 ml of phosphoric acid. Add exactly 50 ml of ferrous ammonium sulphate solution with vigorous stirring. A deep green colour develops when reduction is complete. Titrate the excess of ferrous ammonium sulphate solution with standard potassium permanganate solution, taking the first faint darkening of the clear green colour as the end point. Run a blank, starting from peroxide fusion, in the same manner and at the same time. Note the volumes of potassium permanganate solution required in both cases.

A-4 ESTIMATION OF ACID SOLUBLE CHROMIUM COMPOUNDS IN PIGMENT

A-4.1 Procedure

Weigh accurately about 0.5 to 1 g of the dried pigment of the material sample and digest it in 200 ml of water in a covered beaker. Add 25 ml of sulphuric acid. Boil for 10 min, cool and filter. Then proceed as prescribed in **A-3.1**.

A-5 CALCULATION

A-5.1 Chromium oxide in both the cases as prescribed in **A-3.1** and **A-4.1** is calculated as follows:

Chromium oxide (as Cr_2O_3),

$$\text{percent by mass} = \frac{(A-B)\times N\times 2.534}{M}$$

where,

A = volume of potassium permanganate solution required for blank titration, in ml;

B = volume of potassium permanganate solution required in the assay, in ml;

N = normality of potassium permanganate solution; and

M = mass of the sample taken for the test, in g.

A-5.2 Calculate percent total Cr_2O_3 content of total chromium in pigment as follows:

$$\text{Calculated chromium oxide content in paint} = \frac{100}{\text{(Percent pigment in the sample of paint)}}$$

A-5.3 Calculate acid insoluble chromium oxide as follows:

Acid insoluble chromium (as Cr_2O_3) in pigment, percent by mass = $X - Y$

where,

X = total chromium (as Cr_2O_3) calculated in **A-5.2**, and

Y = acid soluble chromium oxide as determined in **A-5.1**.

ANNEX B

[*Table* 1, *Sl No*. (x)]

TEST FOR RESISTANCE TO PETROL-BENZOL MIXTURE

B-1 REAGENTS

B-1.1 Benzol — Pure

B-1.2 Petroleum Ether — Boiling range 60 to 80°C, aromatic free.

B-2 PROCEDURE

B-2.1 Take a tin plate panel 150 mm × 50 mm × 0.315 mm free from surface imperfections, roughen with No. 0 abrasive paper. Apply the paint by brushing in a single coat to give a dry film mass as specified in IS 101 (Part 3/Sec 4). Allow the paint film to air-dry for seven days followed by keeping it for 24 h under laboratory drying conditions. Then immerse in a mixture of 5 parts by volume of benzol and 95 parts by volume of petroleum ether for 5 min at room temperature not below 15°C. Examine the paint film 30 min after removal of the panel from the solvent mixture.

B-2.2 The material shall be taken to have passed the requirement of this test if the paint film does not show any sign of permanent injury and withstands the scratch hardness test.

ANNEX C

[*Table* 1, *Sl No.* (xii)]

TEST FOR WATER ABSORPTION

C-1 REAGENTS

C-1.1 Paraffin Wax and Beeswax (1 : 1) Mixture — Molten

C-2 PROCEDURE

C-2.1 Take a minimum of 3 panels of reasonably straight grained and well seasoned (to a moisture content of not more than 12 percent) chirwood (*Pinuslongifolia*) approximately 100 mm × 100 mm × 9 mm size, round edges and corners with No. 0 abrasive paper and clean it free from dust and extraneous matter. Apply one coat of the material by brushing so as to completely paint the panels including edges and allow to dry for 24 h at room temperature not below 15°C.

C-2.2 Apply two further coats of the paint in a similar manner and allow the panels to dry for 48 h after the final coat. Seal the edges and corners of the painted panels to a depth of 6 mm all round, by dipping in molten paraffin and beeswax mixture.

C-2.3 Weigh the panel and then completely immerse them in a water-bath maintained at 27 ± 2°C and allow to remain immersed in water for 7 days. Remove the panels from water and absorb the excess moisture by blotting paper and weigh them again in such a manner that the time interval between the withdrawal of panels from water-bath and the weighment is not less than 3 min and not more than 5 min. Note the gain in mass and divide by the total area of the paint film on both sides of the panels not coated with wax-mixture to calculate the results.

ANNEX D

[*Table* 1, *Sl No. (xiii)*]

TEST FOR RESISTANCE TO WARM WATER

D-1 PROCEDURE

Take a mild steel plate, 150 mm × 100 mm × 1.25 mm free from surface imperfections and roughen with No. 0 abrasive paper. Paint the panel and keep it under drying conditions prescribed in **C-2.1**. Immerse the panel in water maintained at 45 ± 2°C for 4 h. Remove the panel, keep at room temperature for 1 h and examine the dried paint film for any fading or change in colour.

ANNEX E

[*Table* 1, *Sl No.* (xiv)]

TEST FOR RESISTANCE TO SALT WATER

E-1 REAGENTS

E-1.1 Calcium Sulphate — Anhydrous.

E-1.2 Magnesium Chloride — Anhydrous

E-1.3 Magnesium Sulphate — Anhydrous

E-1.4 Sodium Chloride

E-2 PROCEDURE

E-2.1 Prepare a panel of mild steel of approximately 150 mm × 150 mm × 1.25 mm size as prescribed in IS 101 (Part 1/Sec 3), and paint with the material by brushing as prescribed in **C-2.1**. Allow the film of the material to air-dry for 24 h at room temperature not below 15°C. Apply another coat of the material over it and allow the panel to air-dry for 48 h. Expose the panel to the following cycle:

a) One hour immersion in salt water of the following composition:
   1) Calcium sulphate, anhydrous — 1.3 g,
   2) Magnesium chloride, anhydrous — 2.6 g,
   3) Magnesium sulphate, anhydrous — 1.7 g,
   4) Sodium chloride — 21.4 g, and
   5) Water to make — 1 000 ml.

b) Three hour exposure outside at an angle of 45° facing south.

c) One hour refrigeration at 4 ± 2°C.

d) Exposure outside at 45° facing south till the commencement of the next cycle.

E.2.2 Repeat the cycle 12 times exposing the same side of the panel during each cycle. Examine the exposed side for signs of breakdown of the paint film.

ANNEX F

[*Table* 1, *Sl No. (xv*)]

TEST FOR DURABILITY

F-1 PREPARATION OF PANEL

Prepare the panel as described in **C-2.1**.

F-2 PROCEDURE

Expose the painted panels, in duplicate, outdoors for 6 months at an angle of 45° facing south. At the end of this period examine the test panels under × 10 magnification. The material shall be taken to have passed the durability test if there is no sign of checking brittleness or other impairment of film integrity, not more than a trace of chalking and no appreciable fading/darkening or change of colour and loss of gloss. In this respect the sample shall not be inferior to the registered sample when tested in the similar manner and at the same time.

NOTE— Provisional acceptance may be given on one month exposure on separate set of painted panels as follows, but the durability of the material be taken only after 6 months exposure as above. The painted panel shall be exposed outdoors for 25 days at an angle of 45° facing south. At the end of this period the panel shall be subjected to a fine spray of water for 6 h at a temperature not above 30°C. A suitable spray is provided by a water supply at a pressure of approximately 13.7 kPa through a rose). The treatment shall be carried out daily for six days. The test panels shall be exposed at an angle of 45° facing south between treatment.

ANNEX G

[*Table* 1, *Sl No.* (xvii)]

DETERMINATION OF CALCIUM COMPOUNDS

G-1 REAGENTS

G-1.1 Dilute Hydrochloric Acid — 1 : 1 (*v*/*v*).

G-1.2 Ammonium Hydroxide — 20 percent

G-1.3 Hydrogen Sulphide Gas

G-1.4 Ammonium Oxalate Solution — Saturated.

G-1.5 Standard Potassium Permanganate Solution — 0.1 N.

G-1.6 Concentrated Nitric Acid

G-1.7 Ammonium Chloride

G-2 PROCEDURE

G-2.1 Weigh accurately about 0.5 g of the extracted pigment in a 250ml beaker. Add 50 ml of dilute hydrochloric acid and boil gently for about 5 min. Dilute the solution to about 200 ml with water and boil again for 2 min. Place the beaker on a water-bath and boil for about 2 to 3 h. Filter the hot solution through a filter paper (Whatman No. 30 or equivalent) and wash the residue with hot water till the filtrate is free from chloride and lead ions.

G-2.2 Treat the filtrate obtained in **G-2.1** with a few drops of dilute nitric acid and boil. Add about 5 g of ammonium chloride and make the solution alkaline with ammonium hydroxide. Boil for 15 min and filter through a filter paper. Wash the precipitate with water and then pass a stream of hydrogen sulphide gas through the filtrate for 15 min. Filter off the sulphides and wash 2 to 3 times with saturated solution of hydrogen sulphide. Boil the filtrate to remove hydrogen sulphide and continue boiling after acidifying the solution. Make the solution alkaline to litmus with ammonium hydroxide and heat to boil. Add sufficient quantity of ammonium oxalate solution and boil for 5 min. Allow the beaker with the precipitate to stand for 2 h. on a water-bath. Filter the residue through a filter paper (Whatman No. 30 or equivalent) and wash several times with hot water. Ignite it in a porcelain crucible to calcium oxide, cool in a desiccators and weigh to constant mass

G-2.3 Alternatively, wash the residue on the filter paper with hot water until the filtrate is free from oxalate ions. Wash down the residue on the filter paper into the original beaker with hot water and wash the filter paper with dilute sulphuric acid collecting the washings in the beaker. Heat to about 70 to 80°C and titrate with standard potassium permanganate solution. When the end point is reached, put the filter paper on which the precipitate was washed in the beaker and complete the titration by adding further quantity of permanganate solution (for filter paper not more than two drops of standard potassium permanganate need be required).

G-3 CALCULATION

G-3.1 Ignition Method

$$\text{Calcium oxide, percent by mass} = \frac{100 \times M_1}{M_2}$$

where,

M_1 = mass of calcium oxide obtained in g; and

M_2 = mass, in g.

G-3.2 Titration Method

$$\text{Calcium oxide, percent by mass} = \frac{2.8 V N}{M_2}$$

where,

V = volume of standard potassium permanganate solution in ml,

N = normality of potassium permanganate solution; and

M_2 = mass of the pigment taken for the analysis, in g.

ANNEX H

[*Table* 1, *Sl No*. (xviii)]

DETERMINATION OF BARIUM SULPHATE

H-1 REAGENTS

H-1.1 Sodium Carbonate — Anhydrous.

H-1.2 Sodium Carbonate Solution — 2 g/l.

H-1.3 Dilute Hydrochloric Acid — 1 : 4.

H-1.4 Ammonium Hydroxide — 20 percent (*m*/*m*).

H-1.5 Concentrated Hydrochloric Acid

H-1.6 Nitric Acid — 1 : 1 (*v*/*v*).

H-1.7 Methyl Orange Indicator Solution — Dissolve 0.1 g of methyl orange in 100 ml of water.

H-1.8 Ammonium Sulphate Solution — 3 percent.

H-2 PROCEDURE

H-2.1 Weigh about 1 g of extracted pigment in a dry and clean 250 ml beaker. Add 20 ml of dilute nitric acid (1 : 1 by volume). Cover the beaker and digest over hot plate for one hour. Dilute the contents to about 150 ml, filter through filter paper, wash the insoluble residue several times. Ignite the residue in platinum crucible. Cool it and add about 8 g of anhydrous sodium carbonate and cover the crucible. Fuse the mixture over a Meker burner for 40 min. The fusion is started with a low flame which is gradually raised to full blast. This precaution is necessary to prevent loss due to overflowing.

H-2.2 Cool and as the melt cools, rotate the crucible so that the fused mass solidifies in a thin layer. This will shorten the time required for leaching. Leach out the fusion with 200 ml of hot water in a 400 ml beaker. Filter through a 15 cm filter paper (Whatman No. 40 or equivalent). Wash several times by decantation, then remove the crucible from the beaker, transfer the insoluble carbonates to the filter and wash with hot sodium carbonate solution, testing after the twelfth washing to be certain that sulphates have been removed completely.

H-2.3 Cover the funnel containing the insoluble carbonates with a watchglass and add hot dilute hydrochloric acid carefully, in small portions at a time to prevent loss, catching the solution in a 600 ml beaker. Add hot dilute hydrochloric acid to the platinum crucible and the beaker in which the leach was made and pour over the filter. Wash the paper with hot water until free from chlorides.

H-2.4 Neutralize this solution with ammonium hydroxide using methyl orange as indicator. Add 0.4 to 0.6 ml of concentrated hydrochloric acid. Dilute to 400 ml with hot water, bring the solution to boiling, and add 25 ml of hot ammonium sulphate solution dropwise with constant stirring to prevent co-precipitation of calcium and magnesium. Transfer the beaker to a warm hot plate and allow to stand for at least 4 h. Filter on an ignited weighed Gooch crucible, wash with hot water several times by decantation. The beaker should be scrubbed thoroughly to remove any adhering precipitate. Continue the washing until free of chlorides. Ignite the crucible in a muffle furnace for 35 min at 850°C. Cool in a desiccator and weigh. Make a blank determination in a similar manner with an equal amount of sodium carbonate and other reagents.

H-3 CALCULATION

$$\text{Barium sulphate, percent by mass} = \frac{100 \times (A - B)}{M}$$

where,

A = mass of the precipitate with the material, in g;

B = mass of the precipitate in the blank determination, in g; and

M = mass of the material taken for the test, in g.

ANNEX J

[*Table*1, *Sl No*. (xxi)]

DETERMINATION OF ACCELERATED STORAGE STABILITY

The paint sample is stored in a closed container and kept at 60°C for 96 h. After the test paint shall not get, liver, curdle or increase in efflux time by more than 8 s, and there shall be no evidence of seeding. The paint shall meet the drying time requirement and shall produce dry film that is uniform in appearance and free from streaking, mottling and seediness. Further, the change in gloss value from the original shall not be more than 5 units.

Bureau of Indian Standards

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Amendments Issued Since Publication

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